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(54)【発明の名称】 X線測定方法及びX線測定装置

(57)【要約】 （修正有）

【課題】 被測定試料の表面に垂直な結晶面について、その結晶配向性やモザイク性、及び面間隔の状態を総合的に把握することができるX線測定方法及びX線測定装置を提供する。

【解決手段】 X線源2と、少なくとも被測定試料10の3方向x，y，zの移動機構と1方向ωの回転機構とを有して成るゴニオメータ装置3と、試料10によって反射されたX線Xdを検出するX線検出器4と、X線源2とX線検出器3との被測定試料10の測定点Mに対する方向θ，2θを変更するX線回転機構とを備えたX線測定装置1を用いて、試料表面10sが1方向ωの回転機構と垂直になるように固定し、試料表面10sで全反射する条件にX線の入射角αを設定する。

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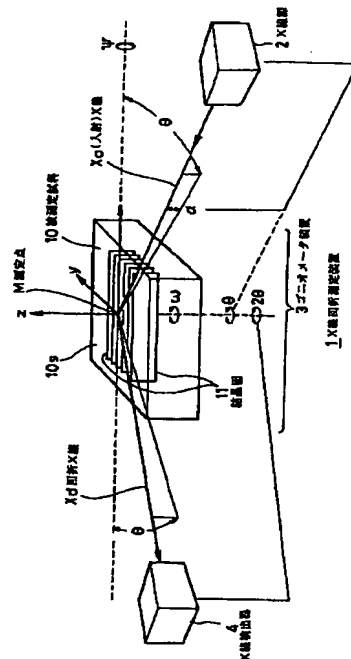
【特許請求の範囲】

【請求項１】 Ｘ線を発生させ単色化させるＸ線源と、少なくとも被測定試料の３方向の移動機構と１方向の回転機構とを有して成るゴニオメータ装置と、

上記被測定試料によって反射された上記Ｘ線を検出するＸ線検出器と、

上記Ｘ線源と上記Ｘ線検出器とをそれぞれ同期させて回転させることにより上記被測定試料の測定点に対する方向を変更するＸ線回転機構とを備えたＸ線測定装置を用いて、

上記被測定試料の表面が上記１方向の回転機構の回転軸と垂直になるように上記被測定試料を固定し、

上記Ｘ線源からのＸ線が上記被測定試料の表面で全反射する条件に該表面への該Ｘ線の入射角を設定し、

上記被測定試料の上記表面に垂直な一結晶面におけるブラッグ反射条件を満たす被測定試料の上記回転機構の回転角及び上記Ｘ線回転機構の回転角とを中心として、上記回転機構の回転角の所定範囲及び上記Ｘ線回転機構の回転角の所定範囲において、

上記被測定試料の上記回転機構と、上記Ｘ線回転機構とのうちいずれか一方の回転機構の回転角を一定値にした状態で他方の回転機構を回転走査させて上記Ｘ線検出器による連続的又は断続的なＸ線測定を行い、

上記一定値を変更して、さらに上記他方の回転機構を回転走査させて上記Ｘ線検出器による連続的又は断続的なＸ線測定を行い、

以降上記一定値の変更と上記連続的又は断続的なＸ線測定を繰り返し、

最終的に上記回転機構の上記回転角の上記所定範囲及び上記Ｘ線回転機構の上記回転角の上記所定範囲内である２次元領域内のＸ線強度分布の測定を行うことを特徴とするＸ線測定方法。

【請求項２】 Ｘ線を発生させ単色化させるＸ線源と、少なくとも被測定試料の３方向の移動機構と１方向の回転機構とを有して成るゴニオメータ装置と、

上記被測定試料によって反射された上記Ｘ線を検出するＸ線検出器と、

上記Ｘ線源と上記Ｘ線検出器とをそれぞれ同期させて回転させることにより上記被測定試料の測定点に対する方向を変更するＸ線回転機構と、

上記１方向の回転機構の回転制御を行う第１の回転制御手段と、

上記Ｘ線回転機構の回転制御を行う第２の回転制御手段とを備えたＸ線測定装置であって、

上記被測定試料の一結晶面におけるブラッグ反射条件を満たす第１の回転制御手段の回転角及び第２の回転制御手段の回転角と、

該第１の回転制御手段の回転角を中心として行われる第１の回転制御手段による回転走査の走査範囲と、

該第２の回転制御手段回転角を中心として行われる第２

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の回転制御手段による回転走査の走査範囲とが制御プログラムに対して与えられ、

上記制御プログラムを用いて、上記第１の回転制御手段及び上記第２の回転制御手段のうちいずれか一方の回転制御手段が上記走査範囲内の一定値である状態に対して、他方の回転制御手段による回転走査及び上記Ｘ線検出器による連続的又は断続的なＸ線測定がなされ、

上記一方の回転制御手段の上記走査範囲内の一定値を該走査範囲内で変更された一定値として、上記他方の回転制御手段による回転走査及び上記Ｘ線検出器による連続的又は断続的なＸ線測定が繰り返され、

最終的に上記第１の回転制御手段による上記回転走査の走査範囲内でかつ上記第２の回転制御手段による上記回転走査の走査範囲内である２次元領域内のＸ線強度分布が得られることを特徴とするＸ線測定装置。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、全反射とブラッグ反射とを利用して行うＸ線測定方法であって、被測定試料の例えば面内結晶配向性や面内モザイク性等の表面特性の評価に用いて好適なＸ線測定方法に係わる。また、このＸ線測定方法に用いて好適なＸ線測定装置に係わる。

【０００２】

【従来の技術】結晶性試料において、その内部位置に応じて結晶方位が変化する様子、例えば結晶配向、モザイク性等を測定する方法としては、被測定試料を回転軸（いわゆるω軸）の回りを回転させながらＸ線の入射角θを変えたときに回折されるＸ線強度を測定するロッキングカーブ法や、被測定試料を回転軸（いわゆるω軸）の回りに回転させながら入射角と回折角とを測定することによって逆格子空間の強度分布を測定する逆格子マッピング法が用いられている。

【０００３】また、結晶内の格子面間隔の状態を評価する方法としては、例えば測定する結晶面からの回折角を測定する２θ−ω法や、（ｈｋｌ）結晶面における反射と（−ｈ−ｋ−ｌ）結晶面における反射のブラッグ条件を満足した結晶方位を正確に走査するボンド法によって結晶面間隔を測定する方法等が用いられている。

【０００４】一方、ω−２θスキャンを各ωにおいて逐次測定することによって、目的とする（ｈｋｌ）結晶面に対する逆格子点ｈｋｌ付近の、被測定試料表面に垂直な逆格子断面強度分布を測定することによって、配向性やモザイク性を測定する手法もある。

【０００５】上述の方法では、被測定試料の表面に対して傾いている結晶面か、或いは被測定試料の表面に平行な結晶面における結晶方位の変化の様子や面間隔を測定して評価を行っている。

【０００６】

【発明が解決しようとする課題】これに対して、被測定

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試料の表面にほぼ垂直な結晶面の場合には、ブラッグ反射した回折Ｘ線が被測定試料の内部に入ってしまうために、前述の方法では回折Ｘ線の測定が不可能である。

【０００７】そこで、Ｘ線の表面に対する入射角αを小さくして、表面でＸ線の全反射を起こさせながら、かつ表面に垂直な結晶面によるブラッグ反射を起こさせることによって（いわゆるＧＩＸＳ：微小入射角Ｘ線回折法）、ロッキングカーブ法や２θ－ω法（又はω－２θ法）による結晶性の評価を行うようにしている。

【０００８】ここで、試料の表面にほぼ垂直な結晶面について、面間隔の異なる領域を含み、結晶方位が結晶表面内で変化する試料の測定においては、前述のＧＩＸＳによるロッキングカーブ法や２θ－ω法を用いて、結晶方位や格子面間隔の変化の様子が測定されている。

【０００９】ところが、ＧＩＸＳによるロッキングカーブ法では、格子面間隔が近くかつ結晶方位がわずかに変化したような試料においては、格子面間隔が近いことによる効果と結晶方位の変化による効果との２つの効果が重なってしまうため、区別して評価することが困難である。

【００１０】一方、ＧＩＸＳによる２θ－ω法では、格子面間隔は区別できるが、結晶方位の僅かな違いによる面間隔の変化を総合的に捉えることが困難である。

【００１１】また、通常の逆格子マッピングにおいては、表面に垂直な方向の逆格子点又は表面に対して斜め方向の逆格子点について、表面に対してほぼ垂直な逆格子断面、場合によっては表面に対して傾斜した断面、を測定するため、表面に平行又は表面に対して傾いた結晶面におけるその結晶面方位の変化を知ることができるが、表面に垂直な結晶面についてはその表面内回転等、結晶面方位の変化を測定することはできない。

【００１２】上述した問題の解決のために、本発明においては、被測定試料の表面に垂直な結晶面について、その結晶配向性やモザイク性、及び面間隔の状態を総合的に把握することができるＸ線測定方法及びＸ線測定装置を提供するものである。

【００１３】
【課題を解決するための手段】本発明のＸ線測定方法は、Ｘ線を発生させ単色化させるＸ線源と、少なくとも被測定試料の３方向の移動機構と１方向の回転機構とを有して成るゴニオメータ装置と、被測定試料によって反射されたＸ線を検出するＸ線検出器と、Ｘ線源とＸ線検出器とをそれぞれ同期させて回転させることにより被測定試料の測定点に対する方向を変更するＸ線回転機構とを備えたＸ線測定装置を用いて、被測定試料の表面が１方向の回転機構の回転軸と垂直になるように被測定試料を固定し、Ｘ線源からのＸ線が被測定試料の表面で全反射する条件に表面へのＸ線の入射角を設定し、被測定試料の表面に垂直な一結晶面におけるブラッグ反射条件を満たす被測定試料の回転機構の回転角及びＸ線回転機構

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の回転角とを中心として、これら２つの回転角の所定範囲において、いずれか一方の回転角を一定値にした状態で他方の回転機構を回転走査させてＸ線検出器による連続的又は断続的なＸ線測定を行い、一定値を変更して、さらに他方の回転機構を回転走査させて上記Ｘ線検出器による連続的又は断続的なＸ線測定を行い、以降一定値の変更と連続的又は断続的なＸ線測定を繰り返し、最終的に２つの回転角の所定範囲内である２次元領域内のＸ線強度分布の測定を行うものである。

【００１４】本発明のＸ線測定装置は、Ｘ線を発生させ単色化させるＸ線源と、少なくとも被測定試料の３方向の移動機構と１方向の回転機構とを有して成るゴニオメータ装置と、被測定試料によって反射された上記Ｘ線を検出するＸ線検出器と、Ｘ線源とＸ線検出器とをそれぞれ同期させて回転させることにより被測定試料の測定点に対する方向を変更するＸ線回転機構と、１方向の回転機構の回転制御を行う第１の回転制御手段と、Ｘ線回転機構の回転制御を行う第２の回転制御手段とを備えたＸ線測定装置であって、被測定試料の一結晶面におけるブラッグ反射条件を満たす第１の回転制御手段の回転角及び第２の回転制御手段の回転角と、第１の回転制御手段の回転角を中心として行われる第１の回転制御手段による回転走査の走査範囲と、第２の回転制御手段回転角を中心として行われる第２の回転制御手段による回転走査の走査範囲とが制御プログラムに対して与えられ、制御プログラムを用いて、上第１の回転制御手段及び第２の回転制御手段のうちいずれか一方の回転制御手段が走査範囲内の一定値である状態に対して、他方の回転制御手段による回転走査及びＸ線検出器による連続的又は断続的なＸ線測定がなされ、一方の回転制御手段の走査範囲内の一定値を走査範囲内で変更された一定値として、他方の回転制御手段による回転走査及び上記Ｘ線検出器による連続的又は断続的なＸ線測定が繰り返され、最終的に第１の回転制御手段による回転走査の走査範囲内でかつ第２の回転制御手段による回転走査の走査範囲内である２次元領域内のＸ線強度分布が得られるものである。

【００１５】上述の本発明によれば、Ｘ線源からのＸ線が被測定試料の表面で全反射する条件に表面へのＸ線の入射角を設定しているので被測定試料の表面に垂直な結晶面におけるブラック反射によるＸ線を測定することができ、被測定試料の表面に垂直な一結晶面におけるブラック反射条件を満たす２つの回転機構の回転角を中心とした所定範囲の２次元領域内のＸ線強度分布を測定するので、被測定試料の表面に垂直な結晶面における格子面間隔の変化及び面内方位の変化の分布を正確に知ることができる。

【００１６】
【発明の実施の形態】まず、図１を用いて本発明に係るＸ線測定方法の概念を説明する。図１中、［１００］・，［０１０］・，［００１］・で示す矢印は、そ

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れぞれｘ軸方向、ｙ軸方向、ｚ軸方向における逆格子空間のユニットベクトルを示す。そして、［１００］＊と［０１０］＊の２つのユニットベクトルが作る平面は、通常ｘｙ平面とされる被測定試料の表面と平行になる。

【００１７】また、図１中、ユニットベクトル［００１］＊に平行な断面Ｓｚは、被測定試料の表面と垂直な逆格子断面である。ユニットベクトル［０１０］＊に平行な断面Ｓｙは、被測定試料の表面と平行な逆格子断面である。

【００１８】前述の従来の測定法では、図中の被測定試料の表面と垂直な逆格子断面Ｓｚにおける逆格子点００１の周辺の回折Ｘ線強度の分布（図中等高線で示す）を測定していた。しかしながら、被測定試料の表面と平行な逆格子断面Ｓｙにおける逆格子点０１０の周辺の回折Ｘ線強度の分布を測定することはできなかった。

【００１９】これに対して、本発明のＸ線測定方法においては、被測定試料の表面と平行な逆格子断面Ｓｙにおける逆格子点０１０の周辺の回折Ｘ線強度の分布を測定することができるものである。

【００２０】続いて、本発明のＸ線測定方法の実施の形態を説明する。図２は、本発明の実施の形態として、本発明のＸ線測定方法及びＸ線測定装置の実施に適用するＸ線回折測定装置１の概略構成図を示す。

【００２１】このＸ線回折測定装置１は、充分単色化されたＸ線Ｘｏを発生し被測定試料１０に入射させるＸ線源２と、被測定試料１０の方位や被測定試料１０内の測定点Ｍの位置等を設定するゴニオメータ装置３と、回折Ｘ線Ｘｄの方向を正確に測定するための分解能を有したＸ線検出器４とから構成される。

【００２２】単色化されたＸ線源２としては、放射光に限らず例えば回転対陰極Ｘ線発生装置やＸ線管球を用いたＸ線発生装置を用い、１つ以上の結晶を用いてＸ線を単色化かつ平行化する機能を有したコリメータ、例えばＧｅ（２２０）結晶面を用いたチャンネルカット結晶２組を用いた４結晶コリメータを備えたモノクロメータによって、Ｘ線を単色化かつ平行化する。

【００２３】尚、モノクロメータに用いられるコリメータとしては、Ｇｅの（２２０）結晶面以外の結晶面を用いたり、Ｇｅ以外の結晶を用いたり、チャンネルカット結晶１つを用いた２結晶コリメータや、単一の結晶面を用いたコリメータ等、その他の単色化を目的としたコリメータを用いることもできる。

【００２４】上述の単色化したＸ線を、スリット等によって被測定試料１０の表面１０ｓの測定位置方向のＸ線のみに制限して、これを入射Ｘ線Ｘｏとして被測定試料１０の表面１０ｓに照射する。

【００２５】Ｘ線源２、スリット等を含むＸ線照射系は、好ましくは傾きを変化させることによって被測定試料１０の表面１０ｓに対する入射Ｘ線Ｘｏの入射角αを変化させることができる構成とする。尚、Ｘ線照射系の

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傾きを変化させる代わりに、ゴニオメータ装置３側の傾きを変化させることによって入射角αを変化させることができる構成としてもよい。

【００２６】ゴニオメータ装置３は、少なくとも被測定試料１０の表面１０ｓにほぼ垂直な回転軸の周囲に被測定試料１０を回転させることができるω回転機構と、被測定試料１０の位置を設定して被測定試料１０の測定したい点を入射Ｘ線Ｘｏの入射位置に移動できるように構成されたｘ軸，ｙ軸，ｚ軸の３軸方向の移動機構とを有して構成される。また、図２のＸ線回折測定装置１では、さらにω回転機構のω回転軸に垂直なψ回転軸によって被測定試料１０の表面１０ｓと平行で被測定試料１０の表面のあおり角を変化させることができるψ回転機構を有して構成されている。そして、好ましくは、これら各回転機構及び移動機構がそれぞれ独立して動くことが可能な構成とする。

【００２７】Ｘ線検出器４は、全体として上述のゴニオメータ装置３の試料回転機構（ω回転機構）と同じ回転軸で回転できる回転機構（２θ回転機構）上に設置する。そして、回折角（２θ）を、ω回転軸やψ回転軸を中心として測定することができるように構成する。

【００２８】また、Ｘ線検出器４の向きは、入射角αに合わせて試料表面１０ｓに対する傾斜角度が入射角に等しい角度αに設定されるようにして、被測定試料１０の表面１０ｓで全反射され、かつ表面１０ｓに垂直な結晶面１１でブラック反射条件で反射された回折Ｘ線Ｘｄを検出できるように構成する。

【００２９】このＸ線検出器４としては、例えばシンチレーション検出器を用いることができ、その前方に回折Ｘ線（散乱Ｘ線）の方位・回折角（２θ）を正確に測定できる機能を有する装置、例えばいわゆるアナライザ装置が設置された構成とする。

【００３０】上述のアナライザ装置としては、例えば１つ以上の結晶を用いたアナライザ結晶を有し、アナライザ結晶は例えばＧｅ（２２０）面を用いたチャンネルカット結晶を用いることができる。尚、アナライザ装置は、例えば３結晶や１結晶のアナライザ装置等の上述の構成以外の構成から成るアナライザ装置を用いることもできる。

【００３１】尚、Ｘ線検出器４は、図２に示す回折Ｘ線Ｘｄ以外のＸ線も検出できるような検出器を用いてもよい。

【００３２】そして、Ｘ線源２とゴニオメータ装置３とＸ線検出器４とは、特定の一点において、本発明に係る被測定試料１０の表面に垂直な結晶面１１の測定の場合においては被測定試料１０の表面１０ｓ上の一点において、Ｘ線源２のθ回転機構のθ回転軸、Ｘ線検出器４の２θ回転機構の２θ回転軸，ω回転軸，ψ回転軸がいずれもこの一点Ｍを通り、かつＸ線源２からの入射Ｘ線がこの一点Ｍに入射するように配置構成される。この被測

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定試料１０の表面１０ｓ上の一点Ｍが、Ｘ線回折強度の測定における被測定試料１０の測定点となる。

【００３３】尚、ψ回転軸は、入射角αの回転軸とω回転軸との交点即ち、上述の測定点Ｍを通らない構成を採ることもできるが、好ましくは図２に示すように上述の測定点Ｍを通る構成とする。

【００３４】また、θ回転機構，２θ回転機構，ω回転機構，ψ回転機構の各回転機構、及びｘ軸，ｙ軸，ｚ軸の各移動機構は、好ましくはコンピュータ制御により、それぞれ独立して、かつ自動的に回転走査又は移動することができ、また任意の角度や任意の位置に設定することが可能な構成とする。

【００３５】尚、θ回転機構と２θ回転機構は、それぞれ同期して回転させることによって、図２に示すように、ψ回転軸から共に回転角θだけ回転させることができる。

【００３６】このようなＸ線回折測定装置１を用いて、本発明に係るＸ線測定方法を実施する場合について説明する。被測定試料１０の表面１０ｓにある測定点Ｍに対して、被測定試料１０の表面１０ｓで全反射を起こすように入射角度αを浅い角度に設定する。

【００３７】その後、被測定試料１０の表面１０ｓにほぼ垂直な結晶面１１によるブラッグ条件を満足するように、被測定試料１０の回転のω回転機構の回転角ωと、検出器４の２θ回転機構の回転角２θとを設定する。ここで、被測定試料１０の表面１０ｓが、ω回転軸と２θ回転軸とを含む面に対して垂直になるように、又は測定する結晶面１１とω回転軸と２θ回転軸が平行になるようにψ回転軸を調整することが望ましい。

【００３８】そして、設定した入射角度αでＸ線Ｘｏを測定点Ｍに入射させると同時に、測定しようとしている結晶面１１例えば（ｈｋ０）結晶面の面内反射のブラッグ反射条件を満足する回転角ω付近において回転角ωを変化させると共に、逐次ω－２θスキャンを行い、回折Ｘ線強度の分布を測定する。即ち、回転角ωを一定値としてω－２θスキャンによる連続的又は断続的なＸ線測定を行ってＸ線検出器４により回折Ｘ線の強度分布を得て、回転角ωを変更して回転角ωを別の一定値として連続的又は断続的なＸ線測定を行い、これを回転角ωの所定範囲内で繰り返す。

【００３９】尚、一定値のω－２θに対してωスキャンを逐次行うことによって、Ｘ線測定を行う方法を採っても同様の結果を得ることができる。

【００４０】これにより、ブラッグ反射条件における回転角ω周辺の、回転角ωの所定範囲内かつω－２θの所定範囲内の２次元領域内における回折Ｘ線Ｘｄの強度分布を得ることができる。即ち逆格子空間における逆格子点ｈｋ０付近の２次元的な強度分布を得ることができる。

【００４１】このとき、結晶面１１の面内反射のブラッ

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グ条件を満足する回転角ωの値、回転角ωを変化させる範囲、及びω－２θスキャンの走査の範囲を設定して測定を行うようにする。

【００４２】そして、好ましくは、結晶面１１の面内反射のブラッグ条件を満足する回転角ωの値、回転角ωを変化させる範囲、及びω－２θスキャンの走査の範囲を与えれば、制御プログラムによって、回転角ω又はω－２θスキャンの内いずれか一方を一定値に固定して、他方を走査して連続的に回折Ｘ線が測定され、一定値の変更と回折Ｘ線の測定が繰り返されて、最終的にこれら回転角ωの範囲内及びω－２θスキャンの走査の範囲内の２次元領域におけるＸ線強度分布が得られるようにＸ線回折測定装置１を構成する。

【００４３】この測定結果を、縦軸をω軸、横軸をω－２θ軸、又はその逆に表示することによって、各々の方向の強度分布を２次元的に把握することができる。尚、この得られた回折Ｘ線強度の分布の測定結果は、縦軸及び横軸を角度の単位から逆格子空間の単位系（長さ分の１）に変換して表示することもできる。

【００４４】上述の測定の手順は、通常の逆格子マッピングによる測定と同様である。

【００４５】上述のように測定を行うことにより、入射Ｘ線Ｘｏの単色性／平行性と、Ｘ線検出器４の精度のよさとを利用して、回折Ｘ線Ｘｄの回折角２θを分解能良く精密に測定でき、逆格子点付近の回折Ｘ線強度分布を２次元的に細かく知ることができる。

【００４６】これにより、通常の逆格子マッピング法の特長を有しているばかりでなく、通常の逆格子マッピング法では測定できない、表面１０ｓにほぼ平行な逆格子点ｈｋ０周辺の表面に平行な逆格子断面における回折Ｘ線強度の分布の測定が可能となる特長を有している。また、前述のＧＩＸＳにおけるロッキングカーブ法では区別が難しかった、格子定数の変化と結晶方位の変化とを区別して２次元的に表示することができる。

【００４７】ここで、得られる２次元の回折Ｘ線強度分布において、ω－２θスキャン方向の広がりや分布は、格子面間隔の変化の様子を表す。また、ωスキャン方向の広がりや分布は、測定した結晶面１１の結晶方位のふらつき分布の様子を表す。

【００４８】そして、結晶方位と格子面間隔の変動が組み合わさったような複雑な構造材料の場合には、ωスキャンを繰り返す、或いはω－２θスキャンを繰り返す、といった１次元的な測定のやり方では、実際の構造とは異なる誤った理解をしてしまう危険性があるが、上述の実施の形態に示す測定方法によれば、網羅的に２次元的な情報が得られるため、このような誤った理解をしてしまう危険を回避することができる。

【００４９】また、表面に平行な逆格子断面Ｓｙを測定することができるために、結晶面１１の面内回転変化の様子を面内格子面間隔とも区別して定量的に測定するこ

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とができる。

【００５０】上述の本発明のＸ線測定方法により実際に測定を行った結果を図３に示す。図３の測定に用いた試料１０は、サファイア基板上に成長させたＧａＮ（窒化ガリウム）エピタキシャル成長結晶である。

【００５１】図３は、この試料１０の表面１０ｓの結晶面１１のブラッグ条件を満たす回転角ω及び回転角２θの周辺で、これらの回転角ω，２θを変化させたときの回折Ｘ線強度の分布を示す。即ち、試料１０の表面１０ｓの面内結晶方位のふらつき分布状態を表している。

【００５２】この測定では、ω－２θ方向にも変化や広がり、即ち格子定数の広がり又は変化は観察されていないが、被測定試料１０の表面１０ｓ内に、又はごく表面近傍に格子定数の異なる例えばＡｌＧａＮ，ＧａＩｎＮ等の物質が混在する場合には、ω－２θ方向にも変化や広がりが観察される。

【００５３】尚、図３では回転角ωの範囲が中心の角度±０．２°、ω－２θの範囲が中心の角度±０．０５°に設定されて測定した場合であったが、これらの測定の範囲は、結晶面間隔の分布や結晶方位の分布等の試料の状態によって、必要に応じて広い範囲或いは狭い範囲を設定して２次元分布測定を行うことができる。

【００５４】上述の本発明に係るＸ線測定方法の応用対象としては、例えばＩＩＩ族元素の窒化物系材料や結晶配向した金属薄膜、半導体薄膜、誘電体薄膜等種々の材料の面内配向の定量的な評価に用いることができる。そして材料の結晶性の正確な評価が可能となり、結晶性の向上に適用して、半導体デバイス、磁気記録デバイス、表示デバイスなど各種デバイスの特性を向上することができる。

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【００５５】本発明のＸ線測定方法及びＸ線測定装置は、上述の実施の形態に限定されるものではなく、本発明の要旨を逸脱しない範囲でその他様々な構成が取り得る。

【００５６】
【発明の効果】上述の本発明によれば、全反射条件とブラッグ条件をほぼ満足させるような条件でマッピング測定することによって、表面にほぼ平行な逆格子断面の強度分布を得ることができ、得られた逆格子断面の強度分布から、被測定試料の表面に垂直な結晶面に対しても、結晶面内方向の結晶方位の変動の様子と格子面間隔の変動の様子を区別して総合的に評価できるようになる。

【００５７】従って、ＩＩＩ族元素の窒化物系材料や結晶配向した金属薄膜、半導体薄膜、誘電体薄膜等種々の材料の結晶性の正確な評価が可能となり、結晶性の向上に適用して、半導体デバイス、磁気記録デバイス、表示デバイスなど各種デバイスの特性の向上に寄与することができる。
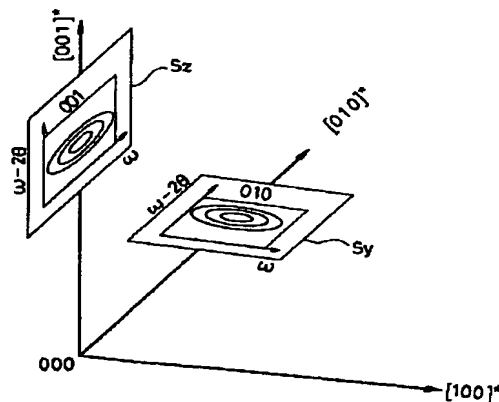
【図面の簡単な説明】
【図１】本発明のＸ線測定方法を説明する逆格子空間の模式図である。

【図２】本発明のＸ線測定方法に用いるＸ線測定装置の概略構成図である。
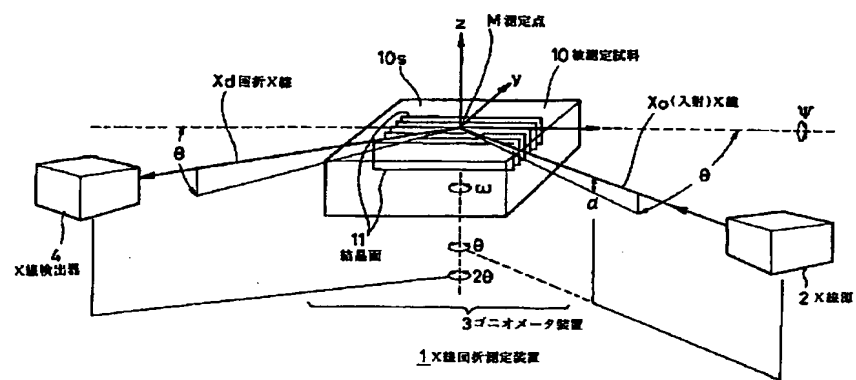
【図３】本発明のＸ線測定方法を用いてＧａＮエピタキシャル成長結晶の表面を測定した測定結果の図である。

【符号の説明】
１　Ｘ線回折測定装置、２　Ｘ線源、３　ゴニオメータ装置、４　Ｘ線検出器、１０　被測定試料、１０ｓ　試料表面、１１　結晶面、Ｘo　（入射）Ｘ線、Ｘd　回折Ｘ線、Ｍ　測定点
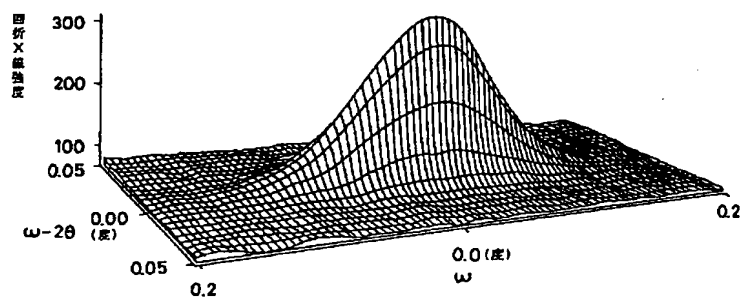
【図１】

【図2】

【図3】

PATENT ABSTRACTS OF JAPAN

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|	
(21)Application number : 10-115360	(71)Applicant : SONY CORP
(22)Date of filing : 24.04.1998	(72)Inventor : MORITA ETSUO

(54) X-RAY MEASUREMENT METHOD AND X-RAY MEASUREMENT DEVICE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an X-ray measurement method and an X-ray measurement device that can totally grasp the crystal orientation property and mosaic property and the state of the interval between surfaces of crystal surfaces that are vertical to the surface of a sample to be measured. SOLUTION: An X-ray source 2, a goniometer device 3 with a traveling mechanism in three directions x, y, and z of at least a sample 10 to be measured and a rotary mechanism in one direction ω, an X-ray detector 4 for detecting X rays Xd being reflected by the sample 10, and an X-ray measuring device 1 with an X-ray rotary mechanism for changing directions θ and 2θ of the X-ray source 2 and the X-ray detector 3 for a measurement point M of the sample 10 to be measured are used, thus fixing a sample surface 10s so that it is vertical to the rotary mechanism of one direction ω and setting an incidence angle α of X rays to conditions for total reflection on the sample surface 10s.
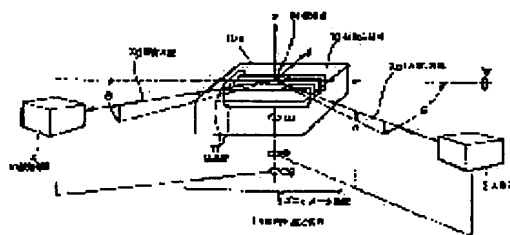
LEGAL STATUS

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than the examiner's decision of rejection or
application converted registration]

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[Number of appeal against examiner's
decision of rejection]

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examiner's decision of rejection]

[Date of extinction of right]

* NOTICES *

CLAIMS

[Claim(s)]
[Claim 1] X line source which is made to generate an X-ray and is made to monochrome-ize, and the goniometer equipment which has the migration device of the three directions of a device under test, and the rolling mechanism of one direction at least, and changes, The X-ray detector which detects the above-mentioned X-ray reflected by the above-mentioned device under test, The X-ray measuring device equipped with the X-ray rolling mechanism which changes the direction over the point of measurement of the above-mentioned device under test by synchronizing the above-mentioned X line source and the above-mentioned X-ray detector, respectively, and rotating them is used. The above-mentioned device under test is fixed so that the front face of the above-mentioned device under test may become vertical to the revolving shaft of the rolling mechanism of the one above-mentioned direction. The incident angle of this X-ray to this front face is set as the conditions in which the X-ray from the above-mentioned X line source carries out total reflection on the front face of the above-mentioned device under test. It centers on the angle of rotation of the above-mentioned rolling mechanism of a device under test which fulfills the Bragg reflection conditions in the 1 crystal face vertical to the above-mentioned front face of the above-mentioned device under test, and the angle of rotation of the above-mentioned X-ray rolling mechanism. In the predetermined range of the angle of rotation of the above-mentioned rolling mechanism, and the predetermined range of the angle of rotation of the above-mentioned X-ray rolling mechanism Where the angle of rotation of one of rolling mechanisms is made into constant value among the above-mentioned rolling mechanism of the above-mentioned device under test, and the above-mentioned X-ray rolling mechanism, carry out the revolution scan of the rolling mechanism of another side, and continuous or intermittent X-ray measurement by the above-mentioned X-ray detector is performed. Change a up Norikazu constant value, carry out the revolution scan of the rolling mechanism of above-mentioned another side further, and continuous or intermittent X-ray measurement by the above-mentioned X-ray detector is performed. henceforth -- modification of a up Norikazu constant value and the above -- the X-ray measuring method characterized by repeating continuous or intermittent X-ray measurement, and measuring X-ray intensity distribution in the two-dimensional field which is above-mentioned predetermined within the limits of the above-mentioned angle of rotation of the above-mentioned predetermined range of the above-mentioned angle of rotation of the above-mentioned rolling mechanism, and the above-mentioned X-ray rolling mechanism eventually.
[Claim 2] X line source which is made to generate an X-ray and is made to monochrome-ize, and the goniometer equipment which has the migration device of the three directions of a device under test, and the rolling mechanism of one direction at least, and changes, The X-ray detector which detects the above-mentioned X-ray reflected by the above-mentioned device under test, The X-ray rolling mechanism which changes the direction over the point of measurement of the above-mentioned device under test by synchronizing the above-mentioned X line source and the above-mentioned X-ray detector, respectively, and rotating them, It is the X-ray measuring device equipped with the 1st roll control means which performs the roll control of the rolling mechanism of the one above-mentioned direction, and the 2nd roll control means which performs the roll control of the above-mentioned X-ray rolling mechanism. The angle of rotation of the 1st roll control means which fulfills the Bragg reflection conditions in the 1 crystal face of the above-mentioned device under test, and the angle of rotation of the 2nd roll control means, this -- with the scanning zone of the revolution

scan by the 1st roll control means performed focusing on the angle of rotation of the 1st roll control means The scanning zone of the revolution scan by the 2nd roll control means performed focusing on the 2nd roll control means angle of rotation is given to a control program. this -- As opposed to the condition that one of roll control means is the constant value in the above-mentioned scanning zone using the above-mentioned control program among the roll control means of the above 1st, and the roll control means of the above 2nd The revolution scan by the roll control means of another side and the continuous or intermittent X-ray measurement by the above-mentioned X-ray detector are made, and the constant value in the above-mentioned scanning zone of the roll control means of the method of up Norikazu as constant value which is in this scanning zone and was changed The revolution scan by the roll control means of above-mentioned another side and the continuous or intermittent X-ray measurement by the above-mentioned X-ray detector are repeated. The X-ray measuring device characterized by acquiring the X-ray intensity distribution in the two-dimensional field which is in the scanning zone of the above-mentioned revolution scan by the roll control means of the above 1st eventually, and is in the scanning zone of the above-mentioned revolution scan by the roll control means of the above 2nd.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention is an X-ray measuring method performed using total reflection and Bragg reflection, is used for assessment of surface characteristics, such as a device under test, for example, the crystal stacking tendency within a field, and mosaicism within a field, and relates to a suitable X-ray measuring method. Moreover, it uses for this X-ray measuring method, and is involved in a suitable X-ray measuring device.
[0002]
[Description of the Prior Art] As an approach of measuring signs, for example, crystal orientation, that crystal orientation changes according to the internal location, mosaicism, etc., in a crystalline sample The rocking curve method which measures the X-ray intensity diffracted when the angle of incidence theta of an X-ray is changed rotating the surroundings of a revolving shaft (the so-called omega shaft) for a device under test, The reciprocal-lattice mapping method which measures the intensity distribution of reciprocal space is used by measuring an incident angle and an angle of diffraction, rotating a device under test around a revolving shaft (the so-called omega shaft).
[0003] Moreover, the 2 theta-omega method which measures the angle of diffraction from the crystal face measured, for example as an approach of evaluating the condition of the lattice spacing in a crystal, the approach of measuring crystal-face spacing by the bond method which scans to accuracy the crystal orientation which satisfied the Bragg condition of the echo in the crystal face (hkl) and the echo in the crystal face (-h-k-l), etc. are used.
[0004] On the other hand, there is also the technique of measuring a stacking tendency and mosaicism by measuring an omega-2theta scan serially in each omega by measuring reciprocal-lattice cross-section intensity distribution vertical to the device-under-test front face near [to the crystal face made into the object (hkl)] reciprocal-lattice point hkl.
[0005] the crystal face to which it leans to the front face of a device under test by the above-mentioned approach -- or it is evaluating by measuring the situation and spacing of change of crystal orientation in the parallel crystal face on the surface of a device under test.
[0006]
[Problem(s) to be Solved by the Invention] On the other hand, on the surface of a device under test, in the case of the almost vertical crystal face, in order that the diffraction X-ray which carried out Bragg reflection may keep close in the interior of a device under test, measurement of a diffraction X-ray is impossible by the above-mentioned approach.
[0007] Then, it is made to perform crystalline assessment by (the so-called GIXS:minute incident angle X-ray diffraction method), the rocking curve method, or the 2 theta-omega method (or omega-2theta law) by making the Bragg reflection by the crystal face vertical to a front face start, making small the incident angle alpha over the front face of an X-ray, and making the total reflection of an X-ray cause on a front face.
[0008] Here, in measurement of the sample from which crystal orientation changes in a crystal front face, the situation of change of crystal orientation or a lattice spacing is measured about the almost vertical crystal face using the above-mentioned rocking curve method and the above-mentioned 2 theta-omega method by GIXS including the field where spacings differ on the surface of the sample.
[0009] However, it is difficult to distinguish and to evaluate by the rocking curve method by GIXS

in a sample from which near and crystal orientation changed [the lattice spacing] slightly, since two effectiveness of the effectiveness by a lattice spacing being near and the effectiveness by change of crystal orientation laps.

[0010] It is difficult to, catch synthetically change of the spacing by the slight difference in crystal orientation on the other hand, although a lattice spacing is distinguishable by the 2 theta-omega method by GIXS.

[0011] Moreover, in order to measure an almost vertical reciprocal-lattice cross section and the cross section which inclined to the front face depending on the case to a front face about the reciprocal-lattice point of the direction of slant in the usual reciprocal-lattice mapping to the reciprocal-lattice point or front face of a direction vertical to a front face, Although change of the crystal-face bearing in the crystal face which inclined to the front face to parallel or a front face can be known, about the crystal face vertical to a front face, the surface internal version etc. cannot measure change of crystal-face bearing.

[0012] The X-ray measuring method and X-ray measuring device which can grasp synthetically the crystal stacking tendency and mosaicism, and the condition of a spacing about the vertical crystal face on the surface of a device under test in this invention for solution of the problem mentioned above are offered.

[0013]
[Means for Solving the Problem] X line source which the X-ray measuring method of this invention makes generate an X-ray, and is made to monochrome-ize, and the goniometer equipment which has the migration device of the three directions of a device under test, and the rolling mechanism of one direction at least, and changes, The X-ray measuring device equipped with the X-ray rolling mechanism which changes the direction over the point of measurement of a device under test by synchronizing the X-ray detector which detects the X-ray reflected by the device under test, and X line source and an X-ray detector, respectively, and rotating them is used. A device under test is fixed so that the front face of a device under test may become vertical to the revolving shaft of the rolling mechanism of one direction. The incident angle of the X-ray to a front face is set as the conditions in which the X-ray from X line source carries out total reflection on the surface of a device under test. It sets in the predetermined range of these two angles of rotation focusing on the angle of rotation of the rolling mechanism of a device under test which fulfills the Bragg reflection conditions in the 1 vertical crystal face on the surface of a device under test, and the angle of rotation of an X-ray rolling mechanism. Where one of angles of rotation is made into constant value, carry out the revolution scan of the rolling mechanism of another side, and perform continuous or intermittent X-ray measurement by the X-ray detector, and constant value is changed. Furthermore the revolution scan of the rolling mechanism of another side is carried out, continuous or intermittent X-ray measurement by the above-mentioned X-ray detector is performed, modification of constant value and continuous or intermittent X-ray measurement are repeated henceforth, and X-ray intensity distribution in the two-dimensional field which is predetermined within the limits of two angles of rotation eventually is measured.

[0014] X line source which the X-ray measuring device of this invention makes generate an X-ray, and is made to monochrome-ize, and the goniometer equipment which has the migration device of the three directions of a device under test, and the rolling mechanism of one direction at least, and changes, The X-ray rolling mechanism which changes the direction over the point of measurement of a device under test by synchronizing the X-ray detector which detects the above-mentioned X-ray reflected by the device under test, and X line source and an X-ray detector, respectively, and rotating them, It is the X-ray measuring device equipped with the 1st roll control means which performs the roll control of the rolling mechanism of one direction, and the 2nd roll control means which performs the roll control of an X-ray rolling mechanism. The angle of rotation of the 1st roll control means which fulfills the Bragg reflection conditions in the 1 crystal face of a device under test, and the angle of rotation of the 2nd roll control means, The scanning zone of the revolution scan by the 1st roll control means performed focusing on the angle of rotation of the 1st roll control means, The scanning zone of the revolution scan by the 2nd roll control means performed focusing on the 2nd roll control means angle of rotation is given to a control program. As opposed to the condition that one of roll control means is the constant value in a scanning zone using a control program among the

roll control means of a top 1st, and the 2nd roll control means The revolution scan by the roll control means of another side and the continuous or intermittent X-ray measurement by the X-ray detector are made, and the constant value in the scanning zone of one roll control means as constant value which is in a scanning zone and was changed The revolution scan by the roll control means of another side and the continuous or intermittent X-ray measurement by the above-mentioned X-ray detector are repeated. The X-ray intensity distribution in the two-dimensional field which is in the scanning zone of the revolution scan by the 1st roll control means eventually, and is in the scanning zone of the revolution scan by the 2nd roll control means is acquired.

[0015] Since the angle of incidence of the X-ray to a front face is set as the conditions in which the X-ray from X line source carries out total reflection on the surface of a device under test according to above-mentioned this invention, the X-ray by the black echo in the vertical crystal face can be measured on the surface of a device under test. Since the X-ray intensity distribution in the two-dimensional field of the predetermined range centering on the angle of rotation of two rolling mechanisms which fulfills the black reflective conditions in the 1 vertical crystal face on the surface of a device under test is measured Distribution of change of the lattice spacing in the vertical crystal face and change of bearing within a field can be known to accuracy on the surface of a device under test.

[0016]

[Embodiment of the Invention] First, the concept of the X-ray measuring method which starts this invention using drawing 1 is explained. Inside of drawing 1 , and [100] *, and [010] * and [001] * The shown arrow head shows the unit vector of the reciprocal space in the direction of a x axis, y shaft orientations, and z shaft orientations, respectively. And [100] * [010] * The flat surface which two unit vectors make becomes the front face of a device under test and parallel which are usually made into xy flat surface.

[0017] Moreover, inside of drawing 1 , and unit vector [001] * The parallel cross section Sz is a reciprocal-lattice cross section vertical to the front face of a device under test. Unit vector [010] * The parallel cross section Sy is a reciprocal-lattice cross section parallel to the front face of a device under test.

[0018] By the above-mentioned conventional measuring method, distribution (a drawing middle quantity line shows) of the surrounding diffraction X-ray intensity of the reciprocal-lattice point 001 in the reciprocal-lattice cross section Sz vertical to the front face of the device under test in drawing was measured. However, distribution of the surrounding diffraction X-ray intensity of the reciprocal-lattice point 010 in the reciprocal-lattice cross section Sy parallel to the front face of a device under test was not able to be measured.

[0019] On the other hand, in the X-ray measuring method of this invention, distribution of the surrounding diffraction X-ray intensity of the reciprocal-lattice point 010 in the reciprocal-lattice cross section Sy parallel to the front face of a device under test can be measured.

[0020] Then, the gestalt of operation of the X-ray measuring method of this invention is explained. Drawing 2 shows the outline block diagram of the X diffraction measuring device 1 applied to operation of the X-ray measuring method of this invention, and an X-ray measuring device as a gestalt of operation of this invention.

[0021] This X diffraction measuring device 1 consists of an X line source 2 which generates X-ray Xo monochrome-ized enough, and carries out incidence to a device under test 10, goniometer equipment 3 which sets up bearing of a device under test 10, the location of the point of measurement M in a device under test 10, etc., and X-ray detector 4 with the resolution for measuring the direction of diffraction X-ray Xd to accuracy.

[0022] the monochromator equipped with 4 crystal collimator using not only synchrotron orbital radiation but 2 sets of channel cut crystals using a collimator with [using the rotating target X-ray generator or the X-ray generator using an X-ray tube / using one or more crystals] monochrome-izing and the parallel-ized function for an X-ray, for example, germanium (220) crystal face, as a monochrome-ized X line source 2 -- an X-ray -- monochrome-izing -- and it parallel-izes.

[0023] In addition, as a collimator used for a monochromator, the crystal faces other than the crystal face (220) of germanium can be used, the crystal of those other than germanium can be used, or the collimators aiming at other monochrome-izing, such as 2 crystal collimator which used one channel

cut crystal, and a collimator using the single crystal face, can also be used.

[0024] A slit etc. restricts the monochrome-ized above-mentioned X-ray only to the X-ray of the direction of a measuring point of 10s of front faces of a device under test 10, and 10s of front faces of a device under test 10 is irradiated by making this into incidence X-ray Xo.

[0025] X-ray illuminating system containing the X line source 2, a slit, etc. is considered as the configuration to which the incident angle alpha of incidence X-ray Xo to 10s of front faces of a device under test 10 can be changed by changing an inclination preferably. In addition, it is good by changing the inclination by the side of goniometer equipment 3 also as a configuration to which the incident angle alpha can be changed instead of changing the inclination of X-ray illuminating system.

[0026] Goniometer equipment 3 has the migration device of 3 shaft orientations of the x axis constituted so that the point of wanting to set omega rolling mechanism which can rotate a device under test 10, and the location of a device under test 10 as the perimeter of a revolving shaft almost vertical to 10s of front faces of a device under test 10 at least, and to measure a device under test 10 could be moved to the incidence location of incidence X-ray Xo, the y-axis, and the z-axis, and is constituted. Moreover, with psi revolving shaft still more nearly vertical to omega revolving shaft of omega rolling mechanism, it is parallel to 10s of front faces of a device under test 10, and it has psi rolling mechanism to which the gate angle of the front face of a device under test 10 can be changed, and consists of X diffraction measuring devices 1 of drawing 2 . And it considers as the configuration which can be preferably moved independently by each [these] rolling mechanism and the migration device, respectively.

[0027] X-ray detector 4 is installed on the rolling mechanism (2theta rolling mechanism) which can be rotated with the revolving shaft same as a whole as the sample rolling mechanism (omega rolling mechanism) of above-mentioned goniometer equipment 3. And an angle of diffraction (2theta) is constituted so that omega revolving shaft and psi revolving shaft can be measured as a core.

[0028] Moreover, according to the incident angle alpha, as whenever [to 10s of sample front faces / tilt-angle] is set as the include angle alpha equal to an incident angle, the sense of X-ray detector 4 is constituted so that diffraction X-ray Xd which total reflection was carried out at 10s of front faces of a device under test 10, and was reflected on black reflective conditions in the crystal face 11 vertical to 10s of front faces can be detected.

[0029] As this X-ray detector 4, a scintillation detector can be used, for example and it considers as the configuration with which the equipment which has the function which can measure bearing and the angle of diffraction (2theta) of a diffraction X-ray (scattered X-rays) to accuracy, for example, the so-called analyzer equipment, was installed ahead [that].

[0030] It has the analyzer crystal using one or more crystals as above-mentioned analyzer equipment, for example, and an analyzer crystal can use the channel cut crystal which used for example, germanium (220) side. In addition, the analyzer equipment which consists of configurations other than the configuration above-mentioned [, such as analyzer equipment of for example, three crystals or one crystal,] can also be used for analyzer equipment.

[0031] In addition, the detector which can also detect X-rays other than diffraction X-ray Xd shown in drawing 2 may be used for X-ray detector 4.

[0032] And the X line source 2, goniometer equipment 3, and X-ray detector 4 In measurement of the crystal face 11 vertical to the front face of the device under test 10 concerning this invention, in one specific point, it sets one on 10s of front faces of a device under test 10. In this one point M, as the incidence X-ray from a passage and the X line source 2 carries out incidence to this one point M, the arrangement configuration also of any is carried out for theta revolving shaft of theta rolling mechanism of the X line source 2, 2theta revolving shaft of 2theta rolling mechanism of X-ray detector 4, omega revolving shaft, and psi revolving shaft. One point M on 10s of front faces of this device under test 10 becomes the point of measurement of the device under test 10 in measurement of X diffraction reinforcement.

[0033] In addition, although psi revolving shaft can also take the configuration which does not pass, Intersection M, i.e., the above-mentioned point of measurement, of a revolving shaft and omega revolving shaft of the incident angle alpha, it is considered as the configuration which passes along the above-mentioned point of measurement M as preferably shown in drawing 2 .

[0034] Moreover, each migration device of each rolling mechanism of theta rolling mechanism, 2theta rolling mechanism, omega rolling mechanism, and psi rolling mechanism and a x axis, the y-axis, and the z-axis is considered as the configuration which it each becomes independent, and it can revolution-scan, can move automatically, and can be preferably set as the include angle of arbitration, or the location of arbitration by computer control.

[0035] In addition, by synchronizing, respectively and making it rotate, theta rolling mechanism and 2theta rolling mechanism can rotate only an angle of rotation theta from both psi revolving shafts, as shown in drawing 2 .

[0036] The case where the X-ray measuring method concerning this invention is enforced using such an X diffraction measuring device 1 is explained. To the point of measurement M in 10s of front faces of a device under test 10, alpha is set as a shallow include angle whenever [incident angle] so that total reflection may be caused at 10s of front faces of a device under test 10.

[0037] Then, the angle of rotation omega of omega rolling mechanism of a revolution of a device under test 10 and angle-of-rotation 2theta of 2theta rolling mechanism of a detector 4 are set up so that the Bragg condition by the crystal face 11 almost vertical to 10s of front faces of a device under test 10 may be satisfied. It is desirable to adjust psi revolving shaft so that the crystal face 11 and omega revolving shaft to measure, and 2theta revolving shaft may be parallel so that 10s of front faces of a device under test 10 may become vertical to a field including omega revolving shaft and 2theta revolving shaft here.

[0038] And while changing an angle of rotation omega in near angle-of-rotation omega are satisfied with of the Bragg reflection conditions of the field internal reflection of the crystal face 11, for example, (hk0), the crystal face, which it is going to measure at the same time it makes point of measurement M carry out incidence of X-ray Xo by alpha whenever [incident angle / which was set up], an omega-2theta scan is performed serially and distribution of diffraction X-ray intensity is measured. That is, the intensity distribution of a diffraction X-ray are acquired with X-ray detector 4, an angle of rotation omega is changed, X-ray measurement continuous as another constant value or intermittent is performed [continuous or intermittent X-ray measurement with an omega-2theta scan is performed by making an angle of rotation omega into constant value,] for an angle of rotation omega, and this is repeated by predetermined within the limits of an angle of rotation omega.

[0039] In addition, by performing omega scan serially to omega-2theta of constant value, even if it takes the approach of performing X-ray measurement, the same result can be obtained.

[0040] Thereby, the intensity distribution of diffraction X-ray Xd in the two-dimensional field of predetermined within the limits of the angle of rotation omega of the angle-of-rotation omega circumference in Bragg reflection conditions and predetermined within the limits of omega-2theta can be acquired. That is, the two-dimensional intensity distribution of the reciprocal-lattice point hk0 neighborhood in reciprocal space can be acquired.

[0041] At this time, it is made to measure by setting up the value of the angle of rotation omega with which are satisfied of the Bragg condition of the field internal reflection of the crystal face 11, the range to which an angle of rotation omega is changed, and the range of the scan of an omega-2theta scan.

[0042] And if the value of the angle of rotation omega with which are satisfied of the Bragg condition of the field internal reflection of the crystal face 11, the range to which an angle of rotation omega is changed, and the range of the scan of an omega-2theta scan are given preferably With a control program, either is fixed to constant value among an angle of rotation omega or an omega-2theta scan. Another side is scanned, a diffraction X-ray is measured continuously, modification of constant value and measurement of a diffraction X-ray are repeated, and the X diffraction measuring device 1 is constituted so that the X-ray intensity distribution in the two-dimensional field within the limits of these angles of rotation omega and within the limits of the scan of an omega-2theta scan may be acquired eventually.

[0043] The intensity distribution of each direction can be grasped two-dimensional by displaying an axis of ordinate on omega shaft, and displaying an axis of abscissa for this measurement result on an omega-2theta shaft or its reverse. In addition, the measurement result of distribution of this obtained diffraction X-ray intensity can also change and display an axis of ordinate and an axis of abscissa on the system of units (1 for die length) of reciprocal space from the unit of an include angle.

[0044] The procedure of above-mentioned measurement is the same as that of measurement by the usual reciprocal-lattice mapping.

[0045] By measuring as mentioned above, using the monochromaticity/parallelism of incidence X-ray Xo, and the merit of the precision of X-ray detector 4, angle-of-diffraction 2theta of diffraction X-ray Xd can be measured with sufficient resolution to a precision, and the diffraction X-ray intensity distribution near a reciprocal-lattice point can be known finely two-dimensional.

[0046] By the usual reciprocal-lattice mapping method, this not only has the features of the usual reciprocal-lattice mapping method, but has the features whose measurement of distribution of the diffraction X-ray intensity in a reciprocal-lattice cross section parallel to the front face of the reciprocal-lattice point hk0 circumference almost parallel to 10s of front faces which cannot be measured is attained. Moreover, distinction can distinguish a difficult change of a lattice constant, and change of crystal orientation, and can express as the rocking curve method in the above-mentioned GIXS two-dimensional.

[0047] Here, in the two-dimensional diffraction X-ray intensity distribution acquired, the breadth of the omega-2theta scan direction and distribution express the situation of change of a lattice spacing. Moreover, the breadth of the omega scan direction and distribution express the situation of wandering distribution of the crystal orientation of the measured crystal face 11.

[0048] And in being the complicated structural material with which fluctuation of crystal orientation and a lattice spacing combined, omega scan repeats or there is a danger carry out an understanding which made the mistake in differing from actual structure, in the way of-like 1-dimensional measurement of repeating an omega-2theta scan, but since two-dimensional information is acquired comprehensively, according to the measuring method shown in the gestalt of above-mentioned operation, it is avoidable in risk of carrying out an understanding which made the mistake in being such.

[0049] Moreover, since the reciprocal-lattice cross section Sy parallel to a front face can be measured, the situation of field internal-version change of the crystal face 11 can be measured quantitatively also [the lattice spacing within a field].

[0050] The result of having measured actually with the X-ray measuring method of above-mentioned this invention is shown in drawing 3 . The sample 10 used for measurement of drawing 3 is the GaN (gallium nitride) epitaxial growth crystal grown up on silicon on sapphire.

[0051] Drawing 3 is the circumference of the angle of rotation omega which fulfills the Bragg condition of the crystal face 11 of 10s of front faces of this sample 10, and angle-of-rotation 2theta, and shows distribution of the diffraction X-ray intensity when changing these angles of rotation omega and 2theta. That is, the wandering distribution condition of the crystal orientation within [of 10s of front faces of a sample 10] a field is expressed.

[0052] although neither change, nor breadth, i.e., the breadth of a lattice constant, or change is observed also in the direction of omega-2theta in this measurement -- the inside of 10s of front faces of a device under test 10 -- or when [from which a lattice constant differs near the front face very much] matter, such as AlGaN and GaInN, is intermingled, for example, change and breadth are observed also in the direction of omega-2theta.

[0053] In addition, although it was the case where the main include angle of **0.2 degrees and the range of omega-2theta were set as the main include angle of **0.05 degrees, and the range of an angle of rotation omega measured, in drawing 3 , according to the condition of samples, such as distribution of crystal-face spacing, and distribution of crystal orientation, the range of these measurement can set up the large range or the narrow range if needed, and can perform two-dimensional distribution measurement.

[0054] As an object for application of the X-ray measuring method concerning above-mentioned this invention, it can use for quantitive assessment of the orientation within a field of various ingredients, such as a nitride system ingredient of an III group element, a metal thin film which carried out crystal orientation, a semi-conductor thin film, and a dielectric thin film, for example. And exact assessment of the crystallinity of an ingredient can be attained, it can apply to crystalline improvement, and the property of various devices, such as a semiconductor device, a magnetic recording device, and a display device, can be improved.

[0055] The X-ray measuring method and X-ray measuring device of this invention are not limited to

the gestalt of above-mentioned operation, and, in addition to this, various configurations can take them in the range which does not deviate from the summary of this invention.
[0056]
[Effect of the Invention] According to above-mentioned this invention, by carrying out mapping measurement of total reflection conditions and the Bragg condition on conditions with which it is made mostly satisfied, the intensity distribution of a reciprocal-lattice cross section almost parallel to a front face can be acquired, and also to the crystal face vertical to the front face of a device under test, the situation of fluctuation of the crystal orientation of crystal-face inboard and the situation of fluctuation of a lattice spacing are distinguished, and it can evaluate now synthetically from the intensity distribution of the obtained reciprocal-lattice cross section.
[0057] Therefore, exact assessment of the crystallinity of various ingredients, such as a nitride system ingredient of an III group element, a metal thin film which carried out crystal orientation, a semi-conductor thin film, and a dielectric thin film, can be attained, and it can apply to crystalline improvement, and can contribute to improvement in the property of various devices, such as a semiconductor device, a magnetic recording device, and a display device.
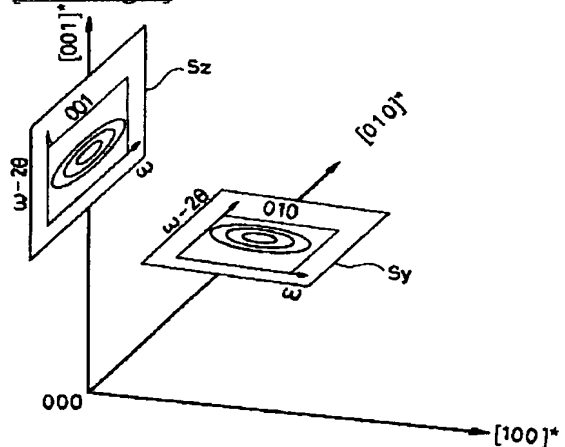
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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the mimetic diagram explaining the X-ray measuring method of this invention of reciprocal space.
[Drawing 2] It is the outline block diagram of the X-ray measuring device used for the X-ray measuring method of this invention.
[Drawing 3] It is drawing of the measurement result of having measured the front face of a GaN epitaxial growth crystal using the X-ray measuring method of this invention.
[Description of Notations]
1 X Diffraction Measuring Device, 2 X Line Source, 3 Goniometer Equipment, 4 X-ray Detector, 10 Device under Test, 10S Sample Front Face, 11 Crystal Face, Xo (Incidence) X-ray, Xd Diffraction X-ray, M Point of Measurement
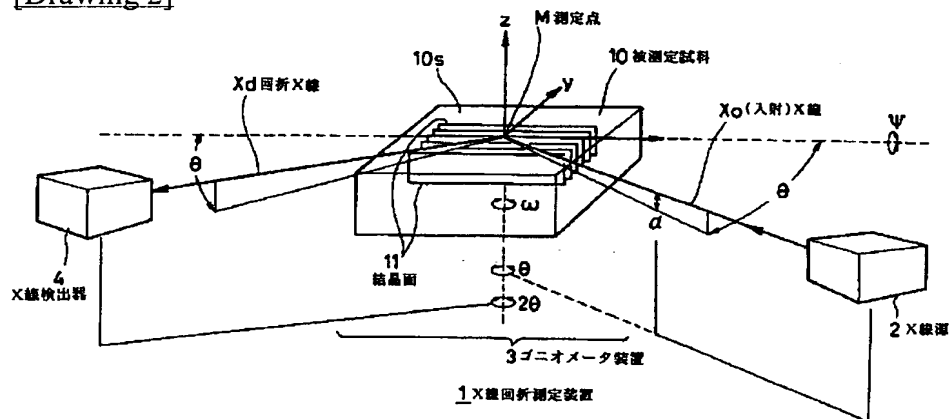

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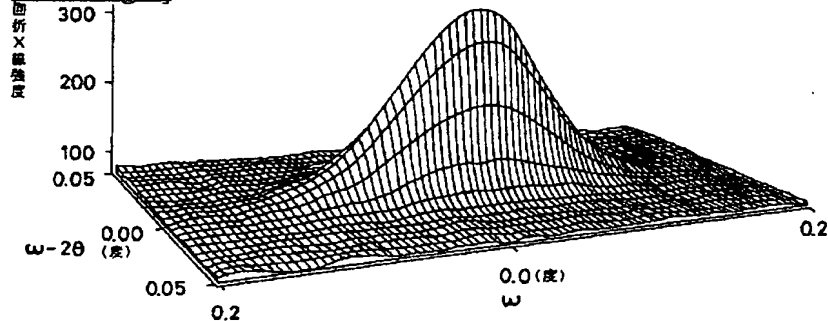3.In the drawings, any words are not translated.

DRAWINGS

[Drawing 1]

[Drawing 2]

[Drawing 3]

[Translation done.]